



Crystals and tablets in the Spanish ecstasy market 2000–2014: Are they the same or different in terms of purity and adulteration?

Claudio Vidal Giné^{a,1,*}, Mireia Ventura Vilamala^{a,1}, Iván Fornís Espinosa^a,
Cristina Gil Lladanosa^a, Nú Calzada Álvarez^a, Ariadna Fitó Fruitós^a,
Joan Rodríguez Rodríguez^b, Antonia Domínguez Salvany^c, Rafael de la Torre Fornell^b

^a Energy Control – Asociación Bienestar y Desarrollo, Spain

^b Human Pharmacology and Clinical Neurosciences Research Group, Institut Hospital del Mar de Investigacions Mèdiques – IMIM, Barcelona, Spain

^c Epidemiology of Drugs of Abuse Research Group, Institut de Recerca Hospital del Mar de Investigacions Mèdiques – IMIM, Barcelona, Spain

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ABSTRACT

Background: Although 3,4-methylenedioxymethamphetamine (MDMA) has a long history in recreational settings, research on its composition (purity and adulteration) has focused only on tablets even though crystal format is readily available for users.

Methods: Drug specimens collected between January 2000 and December 2014 were analyzed at Energy Control's facilities. All samples were voluntarily provided by drug users. Sample identification was made with thin layer chromatography and gas chromatography coupled to mass spectrometry, and quantification with ultraviolet spectrophotometry (only in unadulterated samples).

Results: Between January 2000 and December 2014, 6200 samples purchased as ecstasy by their users were analyzed. Crystals were the most frequent format (60.6%) followed by tablets (38.8%). During the study period, the proportion of samples containing only MDMA was higher in crystals than in tablets. Compared with tablets, adulterated crystal samples contained the same number of adulterants but more combinations of different substances. Although caffeine was commonly detected as adulterant both in crystals and tablets, other substances such as phenacetin, lidocaine, dextrometorphan or methamphetamine were detected almost exclusively in crystal samples. The amount of MDMA in crystal samples remained stable unlike tablets for which a huge increase in MDMA dose was observed since 2010.

Conclusion: Crystal samples of ecstasy showed clear differences compared to ecstasy tablets and this must be taken into account both in research and harm reduction.

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1. Introduction

MDMA (3,4-methylenedioxymethamphetamine) has a long history in recreational settings and several studies have documented its composition in samples sold as ecstasy. In the Netherlands, in over two decades, more than 100,000 samples of controlled drugs have been analysed [1] and several scientific papers about MDMA composition have been published [1–5]. During a period of 16 years, the Drugs Information Monitoring System (DIMS) in the Netherlands analysed the content of 33,006 tablets sold as ecstasy that were handed in by numerous individual

(potential) substance users. Their results showed that the number of high-dose tablets (≥ 106 mg MDMA per tablet) gradually increased from 1998 to 2008. The same holds true for the proportion of tablets that contained only MDMA, reaching the highest levels in 2000 and 2004. After 2004, the purity of ecstasy decreased again, caused mainly by a growing proportion of tablets containing meta-chlorophenylpiperazine (mCPP) [2].

Data about MDMA composition has been also published by the French National Identification System for Drugs and Other Substances (SINTES). Between July 1999 and June 2004, 9453 samples were analysed. Tablets (7004) mainly contained MDMA (82%), and caffeine was the most frequent blended psychoactive substance. Mean MDMA dosage of tablets decreased from 1999 to 2003 [6]. In UK, 101 Ecstasy tablets seized from individuals attending nightclubs were analysed qualitatively to determine if they contained MDMA and quantitatively to determine the MDMA content per tablet [7]. The mean amount of MDMA hydrochloride

* Corresponding author at: Energy Control, Asociación Bienestar y Desarrollo, Quevedo 2 bajos, 08012 Barcelona, Spain. Tel.: +34 952840492.

E-mail address: claudiovidal@energycontrol.org (C. Vidal Giné).

¹ These authors contributed equally to this work.

was 58.7 + 22.9 mg per tablet, with a range of 20–131 mg. The majority (96.0%) of tablets contained less than 100 mg MDMA.

Recently, crystal format has become the most common presentation for MDMA in several European countries [8]. Although the exact reasons for this shift remain unknown, some authors have pointed out towards the decreased purity of MDMA tablets [9] or the convenience of crystal for manufacturers and distributors: the need of a tablet press could be avoided and crystals need less space than tablets to transport the same quantity of the drug [10]. Moreover, this format has prompted new forms of consumption which could imply additional risks to users. Some of these include dabbing MDMA crystal from packets with a moistened finger, making “bombs” out of cigarette papers, and snorting the powder either alone or mixed with other drugs such as cocaine, amphetamines, and ketamine in “designer lines” [9]. But, despite its growing presence in the ecstasy market and potential risks, crystal form has received little to no attention from an analytic or forensic perspective.

On the other hand, adulteration or replacement with other substances to increase economic gain, is common in illegal drugs such as ecstasy [2–4,11,12]. Although in most cases it is a matter of consumer fraud, there are some health related risks that must be taken into account as, for example, the low safety margins of adulterants such as 4-methylamine (4-MA), paramethoxyamphetamine (PMA), and paramethoxymethamphetamine (PMMA). These substances have been sold as MDMA or amphetamine and have also been associated to deaths in several European countries [13,14]. Health risks can be also related to purity, especially when users are unaware of the purity of the drugs they are consuming. This has been the case of ecstasy tablets with high doses of MDMA that can increase the risk of acute toxicity and of neurotoxic harm [15]. These issues, adulteration and purity, highlight the need to gather information about them regarding the crystal form of MDMA because no research has yet been conducted specifically on crystals and whether they differ from tablets.

In this paper, results of the analysis of 6200 drug specimens of ecstasy received in the Energy Control drug testing service over a period of 15 years are presented. Our objective is twofold: on the one hand, to present data about composition, purity and adulteration of MDMA sold in crystal form in the Spanish market and, on the other hand, to show the differences between crystals and tablets in relation to these two indicators. If these differences exist, they should be taken into account when researching ecstasy content in future research.

2. Method

2.1. Sample collection

Drug specimens collected between January 2000 and December 2014 were analyzed at Energy Control's facilities. This Spanish harm reduction project works with recreational drug users. Its drug testing service allows users to submit samples of their drugs to its headquarters to have their contents tested, and to obtain information and advice on risk reduction. Samples were also collected during outreach work in nightlife settings including electronic music festivals, clubs, and underground raves. Information regarding characteristics of the samples and tests results was included in an internal database.

2.2. Laboratory analysis

Sample identification was performed by a combination of validated analysis techniques. For the detection of substances and potentially toxic adulterants, two different chromatographic methods were used: thin layer chromatography (TLC) at the

Energy Control headquarters, and gas chromatography coupled to mass spectrometry (GC/MS) at the IMIM-Hospital del Mar Medical Research Institute in Barcelona (IMIM). For TLC tests, TLC Silica gel 60 F254 (Ref: 1,05554,0001 from Merck) as stationary phase was employed. The TLC plate was developed with three different solvent systems: methanol/25% ammonia solution (100:2.5), methanol, and acetone. After development, analytes were identified comparing their position (retention factor) and colour in the Marquis test with a reference standard. The reference standards were supplied by IMIM.

To confirm TLC results, samples were reanalyzed by GC/MS. From 2000 to 2012 analyses were performed in an Agilent 5890 series II gas chromatograph coupled to a 5971A quadrupole mass spectrometer detector (Agilent). The gas chromatograph was fitted with a 6890 autosampler injector. Samples were injected in split mode into a 5% phenylmethylsilicone column (ULTRA-2, Agilent Technologies), 12 m × 0.2 mm i.d. and 0.33 µm film thickness. The oven temperature was initially maintained at 300 °C for 4 min, the total run time being 14.5 min. Insert liners packed with silanized glass wool were used. The injector and the interface were operated at 280 °C. Helium was used as carrier gas at a flow rate of 0.48 nl/min. The mass spectrometer was operated in electron impact ionization mode at 70 eV. GC/MS was run in scan mode. To identify the substance, retention time was used and to confirm the mass spectra two different libraries were used (2007 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim (Germany) reference library and SWGDRUG MS Library). From 2013 to 2014 ecstasy samples were analyzed using an Agilent 7890B gas Chromatograph coupled to a 5977A quadrupole mass spectrometer detector (Agilent; Santa Clara, CA, USA). The gas chromatograph was fitted with G4513A auto sampler injector. Samples were injected in split mode into a 30 m 0.25 mm i.d., 0.25 µm film thickness 5% phenylmethylsilicone column (HP-5MS, Agilent Technologies). The oven temperature was initially maintained at 90 °C for 2 min and programmed to reach 320 °C at 20 °C per min. It was finally maintained at 320 °C for 9.5 min, the total run time was 21.5 min. Insert liners packed with silanized glasswool were used. The injector and the interface were operated at 280 °C. Helium was used as carrier gas at a flow rate of 1 mL/min. The mass spectrometer was operated in electron impact ionization mode at 70 eV. In order to confirm the mass spectra, four libraries were used: the Searchable Mass Spectral Library NIST/EPA/NIH Mass Spectral Library, Data Version: NIST 14; Searchable Mass Spectral Library Version 2.3 (<http://www.swgdrug.org/ms.htm>), Searchable Mass Spectral Library Cayman Spectral Library (CSL) (<https://www.caymanchem.com/app/template/SpectralLibrary.vm>) and the Energy Control's Mass Spectral library for internal use.

To determine purity, ultraviolet spectrophotometry was performed in a Jenway 6405 apparatus using extinction coefficients. Only unadulterated samples can be quantified by UV.

All statistical analyses were performed using the SPSS 15.0 statistical package.

3. Results

Between January 2000 and December 2014, 6200 samples purchased as ecstasy by their users were analyzed. The most frequent presentation of ecstasy was crystals (60.6%), particularly in the 8 last years evaluated, followed by tablets (38.8%) and, rarely, in other formats such as capsules, gels, paste, liquids, Vaseline, liquorice, and gum (0.6%). Crystals were mostly white whilst tablets varied in colour with different logos and shapes. Here, crystals include powders and crystal solids.

Four sample categories were defined in ecstasy specimens as a function of content: no psychoactive substance (NoPS), only MDMA, MDMA combined with one or more psychoactive

Table 1
Composition, purity and adulteration of ecstasy samples.

	Crystals (n = 3758)	Tablets (n = 2403)
Composition		
Only MDMA	2903 (77.3%)	1362 (56.7%)
MDMA + OPS	251 (6.7%)	171 (7.1%)
OPS	532 (14.2%)	720 (30.0%)
NoPS	71 (1.9%)	151 (6.3%)
Purity in unadulterated samples		
Mean MDMA content	73.5%	86.4 mg
SD	15.8	36.8
Min	3%	5 mg
Max	100%	310 mg
Adulteration		
Adulteration rates	854 (22.7%)	1042 (43.4%)

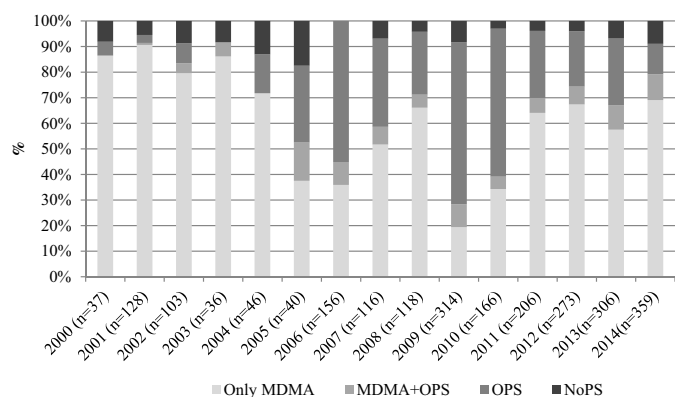


Fig. 1. Percentage of tablets containing only MDMA, MDMA and other psychoactive substances (MDMA + OPS), other psychoactive substances (OPS), or no psychoactive substances (NoPS).

substances (MDMA + OPS), and other psychoactive substances (OPS). In the case of crystals, a high proportion of them contained only MDMA (see Table 1). However, in 2009 this proportion declined until 50% whilst a parallel increase in the number of samples without MDMA but containing other psychoactive substances was observed. Although the same pattern was observed for tablets, the proportion of tablets with only MDMA was lower during the study period (Figs. 1 and 2).

In adulterated crystal samples (MDMA + OPS, OPS and NoPS), 180 different combinations were identified (157 in adulterated tablets). In combinations that included MDMA, the mean number of different substances detected in the samples was 1.2 (SD = 0.6) whereas in combinations without MDMA but other psychoactive substances, the mean number of different substances in the

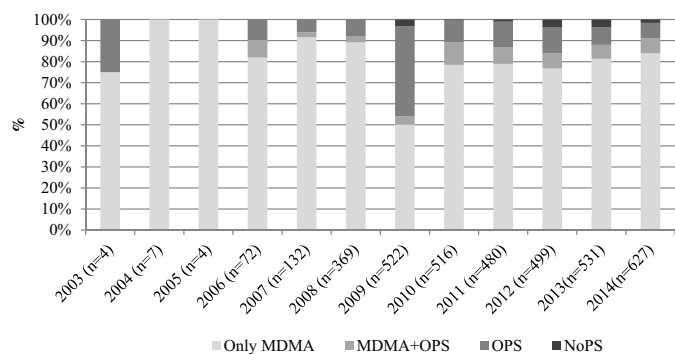


Fig. 2. Percentage of crystal samples containing only MDMA, MDMA and other psychoactive substances (MDMA + OPS), other psychoactive substances (OPS), or no psychoactive substances (NoPS).

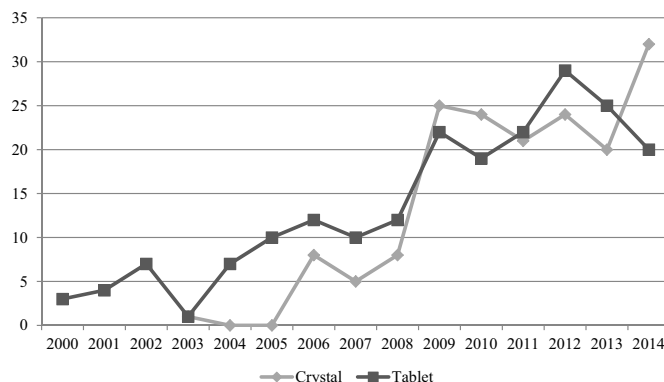


Fig. 3. Number of adulterants identified each year in tablets and crystal samples.

Table 2
Common adulterants found in tablet and crystal samples.

Tablets (n, % ^a)	Crystals (n, % ^a)
Meta-chlorophenylpiperazine (mCPP) (391, 43.9%)	Caffeine (256, 32.7%)
Caffeine (273, 30.6%)	Phenacetin (116, 14.8%)
Metoclopramide (106, 11.9%)	Lidocaine (97, 12.4%)
2,5-dimethoxy-4-bromophenethylamine (2C-B) (48, 5.4%)	Paracetamol (90, 11.5%)
Amphetamine (38, 4.3%)	Dextrometorphan (61, 7.8%)
Paracetamol (33, 3.7%)	Buflomedil (55, 7.0%)
Buflomedil (28, 3.1%)	Procaine (51, 6.5%)
1-(3-trifluoromethylphenyl)-piperazine (TFMPP) (27, 3.0%)	Methamphetamine (48, 6.1%)
3,4-methylenedioxy-N-ethylamphetamine (MDEA) (16, 1.8%)	Meta-chlorophenylpiperazine (mCPP) (34, 4.3%)
Phenacetin (13, 1.5%)	Methylone (23, 2.9%)

^a Percentage of adulterated samples containing the adulterant.

samples was 1.5 (SD = 0.8) (for tablets: 1.2 (SD = 0.5) and 1.4 (SD = 0.7), respectively). Compared with tablets, adulterated crystal samples contained the same number of adulterants but more combinations of different substances. After 2009, the number of adulterated crystal samples declined although the number of different adulterants detected peaked in 2014 (see Fig. 3). The same pattern was observed for tablets.

Caffeine was the main psychoactive adulterant found in adulterated crystal samples followed by phenacetin, lidocaine and paracetamol (see Table 2). Compared with adulterants found in tablets, those found in crystal samples were practically the same although meta-chlorophenylpiperazine (mCPP), other piperazines as BZP and TFMPP and metoclopramide were only identified in a small proportion of crystals. Furthermore, phenethylamines such as 2C-B have been replacing MDMA in tablets since 2008.

In terms of purity, the mean MDMA purity in unadulterated crystal samples was 73.5% (SD = 15.8%). The lowest purity was found in 2009 (3% of MDMA) and the highest in 2010 and 2012 (~100% of MDMA). While the amount of MDMA in crystals remained between 67% and 79% during the study period, in the case of tablets it increased since 2010 reaching a mean dose of MDMA of 113.5 mg in 2014 (see Fig. 4).

4. Discussion

In this paper, data on purity, adulteration, and type of adulterants detected in ecstasy samples obtained from the Spanish illegal market at user-level are presented. To the best of our knowledge, this is the first study documenting these issues for crystal samples of ecstasy and their differences in relation to tablets.

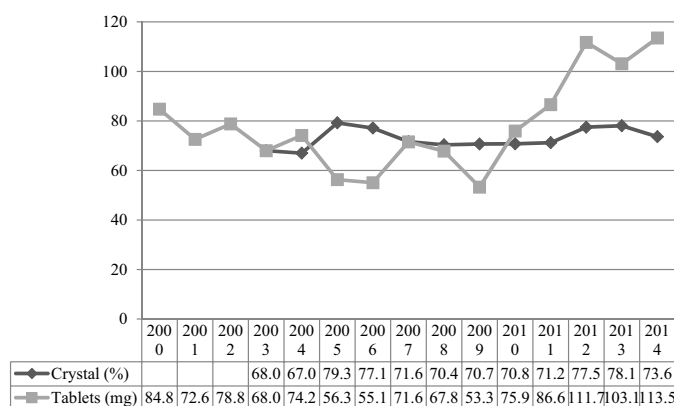


Fig. 4. Amount of MDMA.

The fact that there were more different compositions in crystal samples than in tablets can be explained by the easiness with which crystals can be adulterated. Adulteration in tablets can occur only during the manufacture process while adulteration in crystals is possible at any point of the distribution chain. However, the real reasons underlying the use of the adulterants identified in our study, and not others, remain unknown, except for the case of caffeine whose stimulant effects can compensate an eventual lower dose of MDMA. Our results also confirm that mCPP is the main adulterant in ecstasy tablets, as it has been shown by other researchers in Europe [3]. Furthermore, other piperazines as BZP have also been used as adulterants in tablets [16].

2009 was a year characterized by a shortage of ecstasy on the Spanish market and other European national markets [17,18]. Since then, both crystals and tablets showed an increase in adulteration and the market clearly shifted. As other studies have shown with other markets, supply shortages may produce changes that ultimately affect negatively drug users. The reduction in the availability (and thus purity) of illegal drugs such as ecstasy and cocaine, and the resultant disenchantment among users, was a key motivation for displacement to substituted cathinones, conveniently and legally purchased online [19]. In the case of ecstasy markets, new harms could come from more adulteration and new substances introduced in the market which are also been used as adulterants of ecstasy [20,21].

Our study has showed that the mean purity of MDMA in crystal samples has remained relatively stable over the years in contrast with the variability in MDMA doses of ecstasy tablets which has been also reported by other researchers [2,6,7]. Here, it is worth noting the rise in the amount of MDMA reported in tablets since 2012, as it has also been reported in several European countries [8]. Such high doses can produce significant increases in cardiovascular activity and used in crowded conditions, high ambient temperature, and physical activity (for example, while dancing) they may pose drug users an increased risk of drug toxicity [22]. This issue must be one of the focuses of harm reduction initiatives addressed to ecstasy users in nightlife settings.

A limitation of our study is that sample collection was not probabilistic and depended on the users' request. For this reason, our results cannot provide a full picture of the Spanish drug market. However, our findings are similar to those reported by the Spanish National Toxicology and Forensic Science Institute, a governmental agency responsible for forensic analysis of police seizures in Spain. We both show that ecstasy market in recreational settings is extremely dynamic, that in 2009 there was an increase in the adulteration of products sold as ecstasy, and that, since 2010, there has been an increase in the amount of

MDMA in tablets sold as ecstasy [23]. Other researchers also found similar results when data from user-level and police seizures were compared [1]. But, unlike police seizures data, our results allow us to know the nature and size of the discrepancy between what drug users think they are consuming and what they are actually taking. This has been described as an unique contribution of drug checking services [24].

Because the contents of allegedly MDMA tablets and crystals are mostly unknown for users, scientific knowledge about illegal markets should be shared with them, especially when toxic adulterants or overdosed products are found. In this sense, drug checking services could play an important role in harm reduction. These services create awareness about effects and side effects, educate users about harm reduction strategies and thereby reduce the risks for drug users [25]. Moreover, research in three European cities revealed that integrated drug checking programs do not increase drug use and may even slightly reduce drug use among the target audience [26]. However, there is always a risk that sharing information on composition could provide a marketing tool for drug suppliers and some users could not use it to reduce the risks.

Harm reduction projects and organizations working in nightlife settings are in a pivotal position, not only to establish contact with recreational drug users, but also to collect substances and sound the alert in relation to adulterated or overdosed products sold in local markets. In recent years, a growing number of harm reduction organizations have included drug checking services in their activities and worked in close collaboration with users to share information. The Trans European Drug Information group (<http://www.tedipproject.org/>), created within the framework of the Nightlife Empowerment & Well-being Implementation EU funded project (<http://www.safernightlife.org/>), is an example of this. With this group users have the chance to receive personalized counselling (e.g., dosing and other harm reduction strategies) along with information about their substances. As has been shown by Hungerbuehler et al. [27], through these services it is possible to reach and maintain contact with a hidden population that is not easily accessible by other means and to obtain useful information about it.

On the whole, as our research highlights, there is an urgent need to differentiate between tablets and crystals when researching MDMA composition in products sold on the ecstasy market. As previously mentioned, most forensic research has only been done with tablets when, in fact, users can access both tablets and crystals and clear differences exist between these two different formats of the same substance. It is surprising that research has been unaware of these issues when crystal format exists in the market since at least 2005.

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